APPLICATION NOTE

Analysis of Direct Reduced Iron and Hot Briquetted Iron with ARL EQUINOX 100 Benchtop XRD

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Introduction

Steel is one of the most commonly used materials in human society. Worldwide steel production is still increasing due to growing demand and reached ~ 1550 Mt/y. The majority of raw iron is produced by blast furnaces, which requires high temperatures and high-quality coke. Another process with lower demands in energy and quality of fuel is the Direct Reduction of Iron (DRI), which accounts for ~75 Mt/y of iron. The yielded spongy iron is further processed by sintering to obtain more compact and easily transportable Hot Briquetted Iron (HBI), which is afterwards molten by e.g., arc furnaces to obtain steel alloys. To control the quality of starting iron ore sinters and final DRI or HBI it is necessary to measure the metallization (total iron Fe(total) and metallic iron Fe(metallic)) as well as the Fe^{2+} content. As these values are not accessible by common spectroscopic methods usually, time-consuming wet chemistry is used. Rietveld refinements of easily obtainable X-ray diffraction (XRD) patterns opens a possibility to conveniently determine both FeO(total) content and metallization in a typical process control environment.

Figure 1: ARL EQUINOX 100 X-ray Diffractometer, fast, real-time and convenient.

Instrument

The Thermo Scientific™ ARL™ EQUINOX 100 X-ray Diffractometer employs a custom-designed Cu (50 W) or Co (15 W) micro-focus tube with mirror optics. Such a low wattage system does not require external water chiller or other peripheral infrastructure, allowing the instrument to be easily transported from the laboratory to the field or between laboratories.

The ARL EQUINOX 100 Diffractometer (cf. Figure 1) provides very fast data collection times compared to other conventional diffractometers thanks to its unique curved position sensitive detector (CPS) that measures all diffraction peaks simultaneously and in real time.

Experimental

Fine powdered samples of sinter and HBI were measured during five minutes in reflection geometry using Co Kα radiation. Qualitative and quantitative analysis (Rietveld refinement) were carried out using MDI JADE 2010 with the ICDD PDF4+ database.

Results

Rietveld refinements of sinter (cf. Figure 2) and HBI (cf. Figure 3) yield the amount of the important phases to calculate FeO(total) concentration and metallization in the DRI process (cf. Table 1). The sinter sample contains FeO(total) = 16.5%, while the HBI sample shows Fe(total) = 97.1%, Fe(metallic) = 91.4% (with Fe_3C part, often included in the calculation as metallic Fe) and C = 1.2%. The values are in the range, which are expected for such type of samples.

Figure 2: Rietveld refinement of a sinter sample.

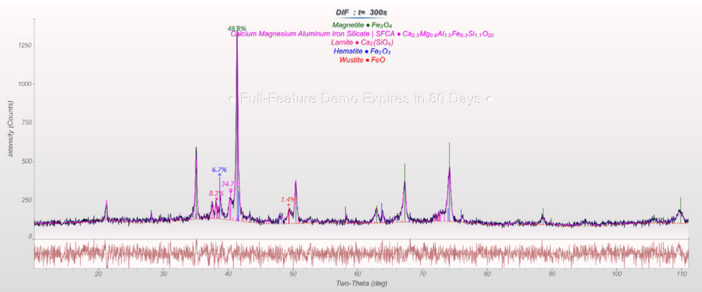

Figure 3: Rietveld refinement of an HBI sample.

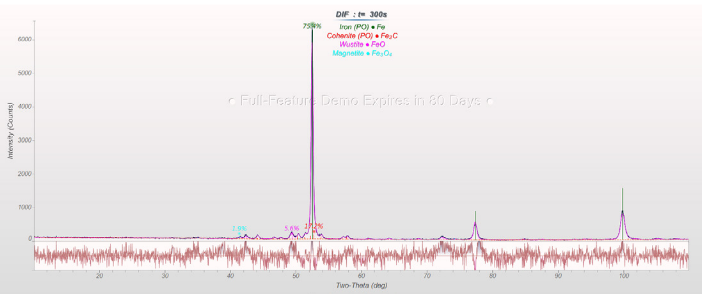

Table 1: Results of the Rietveld refinements of sinter and HBI samples.

Phase (in wt%)	Sinter	HBI
Fe	-	75.4
FeO	1.4	5.6
Fe_3O_4	48.8	1.9
Fe_2O_3	6.7	0
Fe_3C	-	17.2
SFCA*	34.7	-
Larnite (Ca_2SiO_4)	8.3	-

**silico-ferrite of calcium and aluminum*

Conclusion

The ARL EQUINOX 100 Benchtop X-ray Diffractometer in combination with the MDI JADE 2010 software suite and ICDD pdf4+ database is a suitable solution to conveniently determine FeO(total), Fe(total) and Fe(metallic) in sinter and HBI samples. As such analyses are often carried out in production lines for routine operation by the users, an easy-to-use method using a rugged instrument, such as ARL EQUINOX 100 Benchtop XRD, is required. The data processing process can completely be automated to optimize the workflow and reduce potential manual errors, thereby improving the productivity and quality.

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